

Influence of wet-dry and freeze-thaw cycles on the physical and mechanical properties of MICP treated slope soil

Gowthaman, S.

Graduate School of Engineering, Hokkaido University, Sapporo 060-8628, Hokkaido, Japan

Nakashima, K.

Faculty of Engineering, Hokkaido University, Sapporo 060-8628, Hokkaido, Japan

Nakamura, H.

East Nippon Expressway Company Limited, Atsubetsu-ku, Sapporo 004-8512, Hokkaido, Japan

Kawasaki, S.

Faculty of Engineering, Hokkaido University, Sapporo 060-8628, Hokkaido, Japan

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ABSTRACT: Microbial induced carbonate precipitation (MICP) is a bio-mediated soil stabilization method, newly introduced in the field of Geotechnical engineering. Recent investigations have demonstrated that this technique, with several merits, has a significant potential for stabilizing the slope surface. As the MICP treated surfaces are exposed to various weathers including rainfalls, draughts and snowfalls, the durability investigations are requisite prior to the field-scale. The objective of this study is to evaluate the degradation of physical and mechanical properties of the MICP treated slope soil under cyclic wet-dry and freeze-thaw actions. Laboratory experiments were carried out in accordance with the standards. During the tests, mass loss, S-wave velocities and UCS of the specimens were determined. Based on the strength deterioration ratio (SDR), it is demonstrated that the freeze-thaw cycles degrade the physical and mechanical properties more significantly compared to that wet-dry cycles do. Propagation of uneven stresses during the increase in volume of porewater (while freezing) could develop microfractures and ruptures, weakening the properties of treated soil. It is also found that the carbonate content plays more important role in resistance to freeze-thaw more than that of wet-dry cycles. The results would be beneficial at design phase of the treatment, particularly for the considerations on effective life spans.

1. INTRODUCTION

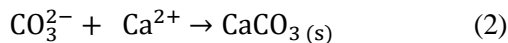
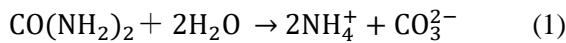
For many decades, stability of slope soils is one of the serious concerns in the field of Geotechnical Engineering. This is mainly because of the fact that sturdiness of most of the transportation structures is highly reliant on the stability of the slopes by which they are supported. During the construction of those structures (e.g. expressways, roads and railways), the natural slopes are modified in a way to fit the construction and design requirements, which often causes the clearance of vegetations and topographical amendments in natural slopes (Guerra et al., 2017; Zhang et al., 2018). Consequently, the slope surfaces become more vulnerable to the climatic measures and associated degradations, posing several risks regarding safety and sustainability.

It is very clear that proper stabilization is essential to a slope to sustain its intrinsic characteristics against the varying nature and accompanied degradation processes. In the past, numerous methods were proposed and investigated to improve the cover condition of slopes. The

most cost-effective and eco-friendliest method for stabilizing the slope is establishment of vegetation such as grass, weeds, plants and etc. (Chirico et al., 2013). The vegetation as an engineering material, serves as a crust layer between atmosphere and surface, enduring the slope from the direct contact of climatic measures such as rainfalls, snowfalls, etc. Also, their roots reinforce the surface, enhancing the stability of slope substrates. However, to achieve the fullest benefits, it takes quite a long time, and the growth and survival of vegetation are limited to certain regions such as subarctic and arid zones (Jiang et al., 2019). Some of other methods widely in application stage are mechanical compactions, geosynthetic applications and chemical stabilizations (Gowthaman et al., 2019a; Kumar and Das, 2018). Majority of these stabilization techniques utilize mechanical energy and artificial materials, both of which required substantial energy for manufacture and installation. Chemical grouts (such as cements, epoxy, acrylamide, phenoplasts, silicates, and polyurethane) are also found to be reliable, could enhance the surface by filling pore spaces and binding soil particles (Arya et al.,

2018; DeJong et al., 2010). However, almost all these materials are reported to be toxic and hazardous, causing pollutions and influx of dangerous substances to the geo-environment; therefore, their applications to the field-scale are extensively under the scrutiny of public policy (DeJong et al., 2010; Gowthaman et al., 2018). Confluence of these drawbacks in the conventional methods necessitates the exploration and development of new methods for stabilizing the slope soil more effectively.

As an emerging technique, MICP (microbial induced carbonate precipitation) has drawn substantial attentions in the past decade. MICP is a biochemical process which persuades the calcium carbonate bio-cement within the embedded soil matrix as a consequence of metabolic activity. The process involves two prime biochemical reactions (Eq. 1 and Eq. 2). First, the urease produced by soil bacteria catalyzes the hydrolysis of urea (Eq. 1). At the supply of calcium ions, calcium carbonate precipitates at nucleation sites provided by the bacterial cells (Eq. 2), which bonds the soil particles i.e. acting in a way similar to that of bridges among soil particles (Achal and Kawasaki, 2016; DeJong et al., 2010; van Paassen et al., 2010), enhancing the engineering characteristics.



In the recent past, few studies have demonstrated that the MICP can be potentially applied to stabilize the slope soil (Gowthaman et al., 2019b, 2019c; Jiang et al., 2019). Similar to the conventional grouting techniques, MICP has also been proposed for surficial treatment (Gowthaman et al., 2019b, 2019c), which tends to promote the cover condition of the slope, so that to withstand various degradation processes. Rainfall induced surface erosion is one of the factors threatening the stability (Zhang et al., 2018). By investigating sets of MICP treated slope models, Jiang et al. (2019) have recently concluded that treating the slopes by 1 mol/L cementation solution would give optimum resistance against erosion induced by artificial rainfall. It is worth noting that despite of the considerable interests on MICP, the available information on the durability and performance are very limited.

Processes of wet-dry and freeze-thaw are also other serious concerns in Geotechnical Engineering, affecting the long-term performances. After the treatment, sooner or later, the slope surfaces are exposed to varying climates and environment conditions. During the rainfalls and subsequent evaporations, surfaces are subjected to periodical wet-dry process (Tang et al., 2016), which might cause progressive damages in MICP surfaces. Also, the subarctic regions like Hokkaido, Japan typically experiences long severe winters. Based on the 20 years of

meteorological data, soils in Hokkaido freezes for more than 100 days per winter (Farukh and Yamada, 2018). During the thawing in early spring, infiltration of meltwater is impeded by the frozen soil, which in turn to the generation of surface runoff, leading to the detachment of soil from thawed surface (Kværnø and Øygarden, 2006). However, the effects of wet-dry and freeze-thaw have not been extensively investigated in the past. Therefore, the main objective of this study is to investigate the influence of variable wet-dry and freeze-thaw cycles on the physical and mechanical characteristics of MICP treated slope soil, representing the erosion prone expressway slope in Hokkaido, Japan.

2. MATERIALS AND METHODS

2.1. Slope soil and bacteria

One of the erosion prone expressway slopes, Onuma (Hokkaido, Japan), was investigated in this research work. The soil was collected from the expressway slope and transported to the laboratory. According to the AASHTO (American Association of State Highway and Transportation Officials) soil classification system, the slope soil can be categorized as A-3 (fine sand) with the average particle size of 0.23 mm.

In the MICP technique, using indigenous bacteria has several merits over applying exogenous bacteria, such as elimination of microbial pollution, sustainment of enzymatic capabilities and overcoming of ambiguities regarding regulatory acceptance (Gomez et al., 2018). Therefore, the native ureolytic bacteria were isolated from the slope soil and were augmented back to treat the slope soil. The isolation process of the bacteria can be found in the previous work (Gowthaman et al., 2019b).

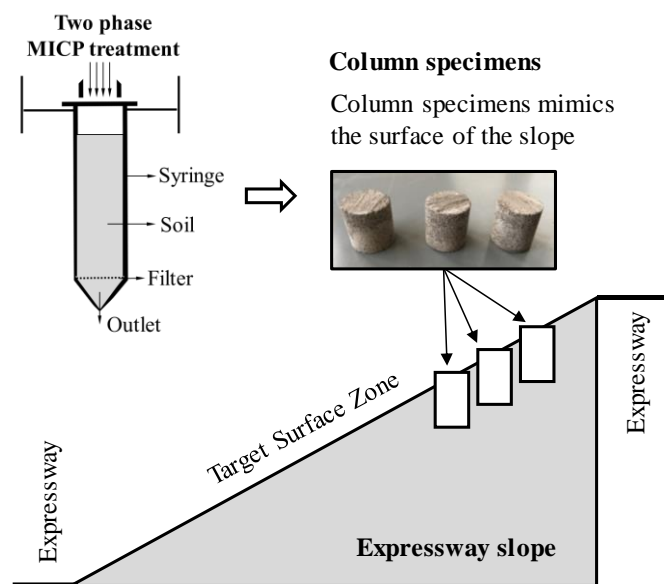


Fig. 1. Conceptual illustration of MICP treatment and the simulation of field-scale (slope surface) by laboratory-scale (columns specimens)

The bacteria used in this work are *Lysinibacillus xylanilyticus*, gram positive strains, which show the optimum urease activity of around 2 – 3 mmol/min/mL at 25°C (Gowthaman et al., 2019c).

2.2. MICP treatment and reagents

The columns specimens were prepared using syringe molds (diameter of 3 cm, height of 6 cm) at a relative density of around 60%. As conceptually illustrated in Fig. 1, the column specimens are considered to be simulating the slope surface (real field) to be treated. The MICP treatment was performed to the soil by using a simple two-phase surface percolation technique as proposed in the previous work (Cheng and Cord-Ruwisch, 2014). The MICP set up is also graphically depicted in Fig. 1, together with the image of treated columns. At the first phase, the bacteria which had been cultivated in NH₄-YE medium (ammonium sulfate 10 g/L, tris buffer 15.7 g/L and yeast extract 20 g/L) under shaking incubation (25°C, 160 rpm) were harvested at the OD₆₀₀ of 4 – 4.5, and applied on the surface of the specimens to percolate under the gravitational and capillary forces. Around 2 – 3 hours were given to a better immobilization of bacteria cells with soil grains. At the second phase, the cementation solution comprised of CaCl₂ (111 g/L), urea (60 g/L) and nutrient broth (6 g/L) was injected under the similar free-drained conditions. The treatment was performed for 14 days, in which the cementation solution was injected every 24 hours. As the performance of the bacteria decrease gradually with the time (typically become insignificant after 7 – 8 days) (Martinez et al., 2013), the biological injection was once again performed after 7 days of the treatment.

In this study, different treatment levels were achieved by controlling the number of cementation solution injections. Around 13%, 17% and 22% of CaCO₃ (by weight) were achieved by 7, 10 and 14 injections of cementation solution respectively. The CaCO₃ content precipitated in the specimens were measured by typical acid washing method (Fukue et al., 2011), and the carbonate contents are presented as the ratio between the weight of precipitated carbonate and the weight of the uncemented dry soil.

After the treatment process, the samples were rinsed with enough distilled water to remove the unreacted salts. Finally, the molds were cut, taken out, and carefully trimmed to make the surfaces flat and smooth before subjected to the wet-dry and freeze-thaw experimentations. Two different sets of specimens treated identically were separately experimented for wet-dry and freeze-thaw tests.

2.3. Wet-dry (WD) test

As there is no standards available for bio-cemented specimens, WD test was performed according to the standard suggested to the conventionally cemented soils

(ASTM D559-03, 2003). During the wetting process, specimens were inundated at room temperature (25 ± 1 °C) for 6 hours, followed by the oven dry at 70 ± 1 °C for 48 hours (drying process). Specimens were subjected to the total number of fifty WD cycles.

2.4. Freeze-thaw (FT) test

FT tests were conducted according to the standard recommended for durability of rock specimens for erosion control under freezing and thawing conditions (ASTM D5312-92, 1997). Initially, all the specimens were saturated well in the distilled water for 12 hours. Afterwards, they were subjected to freezing at the temperature of -20 ± 1°C for 12 hours, followed by the thawing at 25 ± 1°C for 12 hours. Throughout the experiment, the sample conditions were maintained to be fully saturated.

2.5. Evaluation of specimens

During both WD and FT experimentations, the physical and mechanical changes of specimens were determined by measuring mass loss and S-wave velocity (SonicViewer-SX: 5251, Japan). The uniaxial compressive strength (UCS) tests were performed to the specimens before and after subjected to the cyclic tests by using needle penetrometer (JGS 3431-2012, 2012). The needle penetration test is an International Society for Rock Mechanics (ISRM) recommended method for determining UCS of soft, weak to very weak rocks and cemented soil specimens.

3. RESULTS AND DISCUSSION

3.1. Aggregate stability

The physical damage during cyclic tests was evaluated by the mass loss of the specimens. The mass loss was carefully measured after every 5 cycles with neither any structural damages nor considerable changes in temperature. Fig. 2 shows the mass loss of the specimens subjected to cyclic WD and FT actions. The results suggest that the mass loss by the end of 50 WD cycles is less than 5% in all three levels of cementation, and the influence of precipitated calcium carbonate content against WD effects is likely to be not very significant. On the other hand, the FT results reveal that the level of cementation remarkably influences the physical damage of the specimens subjected to FT cycles. For example, by the end of 25 FT cycles, the mass loss in specimens cemented to 13% by calcite is around 50%, experienced a severe structural damage with the separation of fragments. In contrast, average mass loss is around 3% in the specimens cemented to 22% by calcite, which underwent only a minor surficial damage.

It is clearly perceived that the degradation mechanisms of MICP specimens under WD and FT cycles are different. During the WD test, regardless of cementation levels, rate

of mass loss was high at the beginning (within couple of WD cycles) and remained relatively stable during subsequent cycles. This could possibly be attributed to the suspension of feeble deposits of calcium carbonates. During the MICP treatment, the calcium carbonate is precipitated in various forms such as primary bonds (strongly forms at particle contacts), individual crystals and accumulation (on the grain surface), amorphous and powdery deposits (Lin et al., 2016; Wang et al., 2019). In fact, these powdery bonds are often formed when the carbonates are deposited at its early stage of the crystallization. When the specimens are submerged during the wetting, the water penetrates the specimen and drives the powdery deposits to fall into suspension. This corrosion mechanism is similar to that reported to the calcarenite rocks (Ciantia et al., 2015).

The MICP treated soils have porous structure. When the specimens are subjected to freezing (during FT cycles), water turns to ice, which increases the water volume by up to 9% (Bell, 1992), resulting the development of uneven stresses around the soil particles. In fact, the resultant stress acting on soil particles are distributed to the connections around them i.e. to the calcium carbonate bonds. When the acting tensile stresses exceed the maximum tensile strength of carbonate bonds, the bonds would start cracking. For the specimens cemented to 13% carbonate, the bonds were weak, resulted the highest disintegration of aggregates (around 50%). But, for the specimens treated to 22% carbonate, the contact strength could be adequate to resist the stresses developed during FT, resulted negligible loss of aggregates (around 3%). In the point of view of erosion, neither aggregate collapse nor fragmentation are resulted by cyclic WD effects. But conversely, severe erodibility of MICP slope soil is evidenced under cyclic FT effects. However, the FT induced erosion could be controlled by treating the soil

with appropriate quantity of calcium carbonate cement. It is also clear that higher the level of cementation, higher the resistance to the FT induced erosion.

3.2. Mechanical characteristics

The influence of WD and FT actions on the mechanical behavior of MICP specimens was evaluated through S-wave velocity and UCS. Previous researchers have demonstrated that the S-wave velocity is a credible measure which can be used to assess particularly the particle connections in MICP specimens (Feng and Montoya, 2017). Table 1 presents the S-wave velocity values of the specimens before and after subjecting to WD and FT cycles. Overall, it can be seen that the S-wave velocity reduced significantly in both WD and FT actions, suggesting that particle connections have been deteriorated in both cases. It should be noted that the S-wave velocity of untreated slope soil ranges between 150 and 200 m/s. When the soil particles are connected by calcium carbonate bio-cement, the transmission of S-waves increases, leading to the increase in S-wave velocity values with respect to the level of cementation. However, when the connections are degraded and/or deformed under the both cyclic treatments, the velocity values tended to decrease.

The UCS measurements, illustrated in Fig. 3, also show the similar tendency of S-wave velocity. It can be observed that the decrease in UCS values is lower when exposed to WD cycles (Fig. 3(a)) compared to that under FT cycles (Fig. 3(b)). As explained in the previous section, the corrosion of powdery and weak deposits caused the mass loss, and that would also considerably affect the mechanical responses. Regardless of weak or strong, if the carbonates deposit at or near particle contacts, they contribute to the strengthening of MICP soil skeleton by supporting primary bonds.

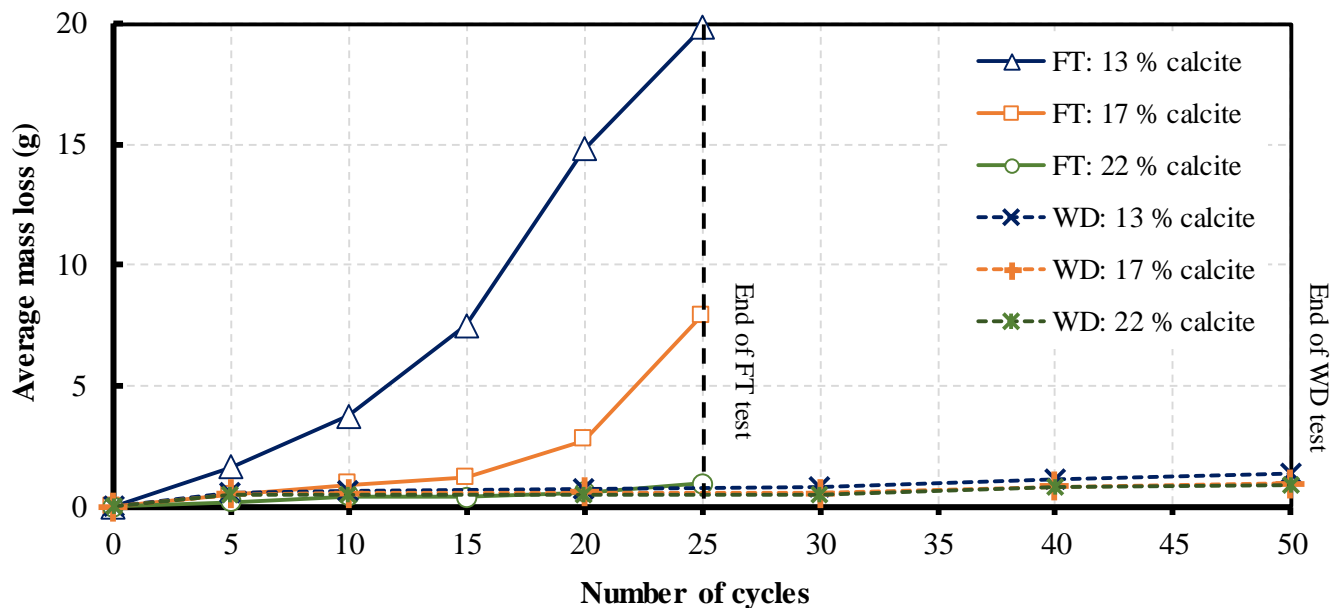


Fig. 2. Average mass loss of the specimens subjected to cyclic treatments (WD and FT)

Table 1. The S-wave velocity values before and after subjected to WD and FT cycles

Average S-wave velocity (m/s)	50 WD cycles		25 FT cycles	
	Before	After	Before	After
1150	810	1040	< 800	
1350	1060	1240	950	
1690	1260	1500	1390	

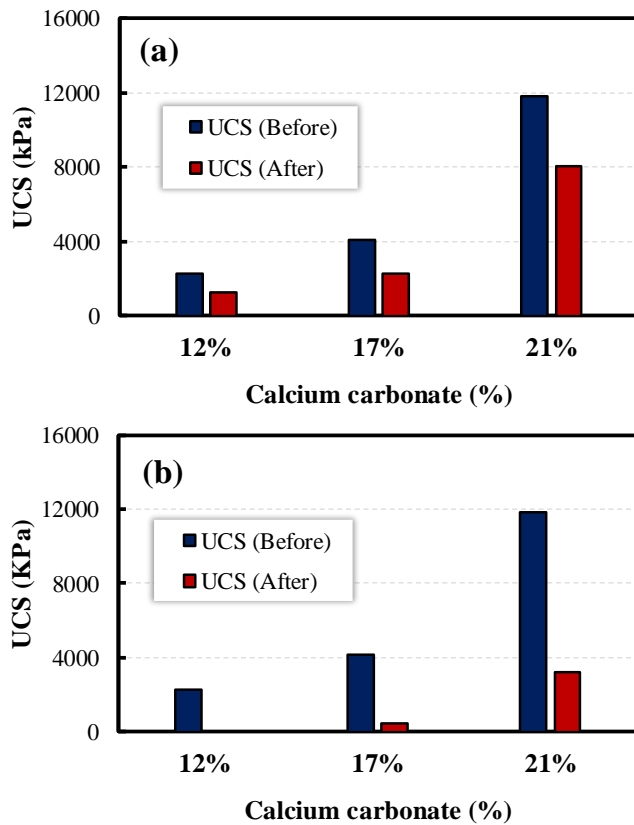


Fig. 3. The variation of UCS before and after the exposure of cyclic (a) WD tests and (b) FT tests

When the suspension of those weak deposits occurs, there would be a weakening in primary chains in soil skeleton, leading to the decrease in UCS and S-wave velocities (Fig. 3(a) and Table 1 respectively).

Mechanical damage of MICP specimens appears to be higher under the exposure to FT cycles (Fig. 3(b)). As explained in the previous section, when the porewater undergoes freezing process, uneven tensile stresses are developed around the soil particles, leading to the formation and the growth of microcracks. Consequently, the MICP skeleton gets weakened, could be able to withstand only lower compression stresses compared to that of unexperimented. In fact, the resistance to the FT damage depends on the porosity of the soil matrix. Higher porosity permits more and rapid transfer of water through the soil matrix, which could aid to prevent the damages FT effects. However, in the case of MICP, soils of less

porosity i.e. fine-grained soils were more durable against the FT actions compared to that of coarse-grained soils i.e. soils of higher porosity (Cheng et al., 2016). This is due to the fact that the number of contacts of each soil grain increase with the decrease in porosity. In the case of fine-grained soils, higher number of particle contact points facilitated to increased MICP bridges, which reduce the acting tensile stresses per connection, withstanding long FT cycles. The soil investigated herein also fine-grained soil. And, the specimens were investigated herein up to its possible optimum cementation level in order to assess the fullest feasibility. It also should be noted that when the specimens were treated over 22 – 23% by calcium carbonate, bio-clogging stage was frequently achieved similar to that reported by (Cheng and Cord-Ruwisch, 2014).

3.3. Strength deterioration ratio (SDR)

In order to better understand the effect of precipitated calcium carbonate content against mechanical degradation, *SDR* values under cyclic treatments are computed by using the following relationship (Eq. 3).

$$SDR = 1 - \frac{UCS_f}{UCS_i} \quad (3)$$

SDR is the simplified measure, typically used to explain the deterioration of specimens exposed to cyclic treatments and had been broadly used by researchers for soft to hard rock materials (Hale and Shakoor, 2003; Khanlari and Abdilor, 2015). The UCS_f represents the compressive strength at the end of the cyclic treatment and UCS_i represents the compressive strength of virgin MICP specimens. The *SDR* varies between “0” and “1”. A rating of “1” is allotted to the weakest specimen, reveals complete degradation at the end of the cyclic treatment. For the strongest specimens, which do not show any damages after the treatment, a rating value of “0” is allotted.

The calculated *SDR* values of the samples subjected to WD cycles are plotted in Fig. 4(a), together with the *SDR* values of various sandstones material obtained from the literature (Khanlari and Abdilor, 2015). The SDR_{WD} values of sandstones (subjected to 30 WD cycles) range between around 0.1 and 0.2. Comparatively, MICP specimens show higher deterioration under WD cycles (SDR_{WD} of 0.3 – 0.45), suggesting that MICP cemented soils are more susceptible than sandstones. In fact, as suggested by Li et al. (2019), the fatigue stresses developed during the wet-dry cycles could affect the mechanical responses of cemented specimens. In the case of MICP, the thermal expansion coefficients of crystallized calcium carbonate and soil particles are different, leading to the development of irregular internal stresses during the cyclic wet-dry process, which possibly caused the deformations of particle contacts hence deteriorated the UCS of MICP treated slope specimens.

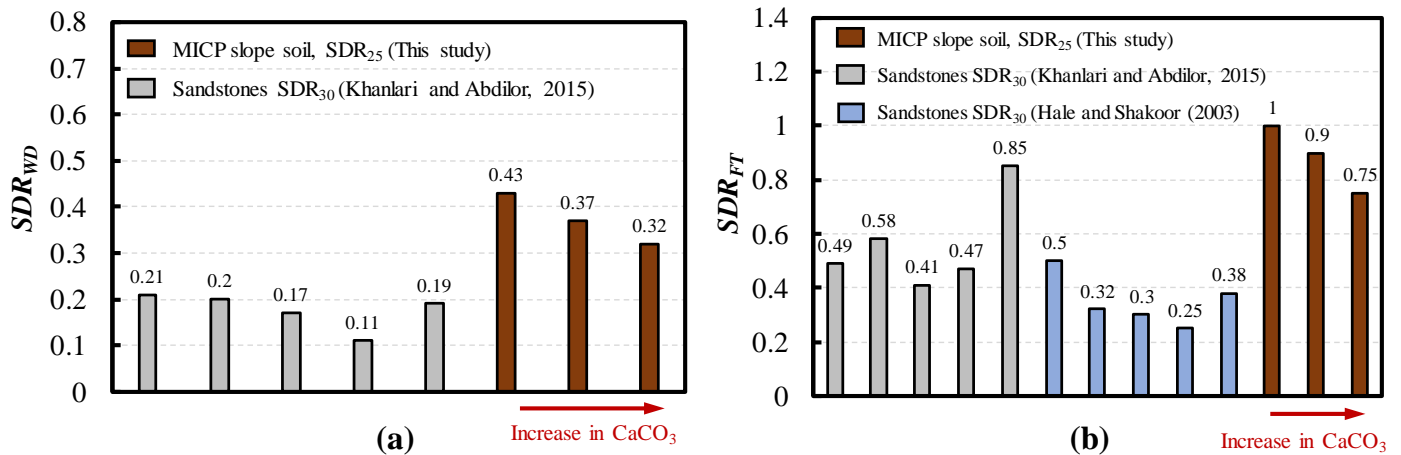


Fig. 4. SDR values of the MICP specimens subjected to (a) WD cycles and (b) FT cycles, compared together with the SDR values of various sandstone materials.

However, the SDR_{WD} values considerably decrease with the increase in calcium carbonate content (Fig. 4(a)). The observation demonstrated that the high deposition of carbonates likely to be preserving the MICP responses against WD actions. Longer MICP treatments achieve high precipitation content, which could strengthen the primary connections, providing high resistive forces during the development of uneven fatigue stresses.

Fig. 4(b) presents the SDR values of specimens under FT action. Comparing the SDR_{FT} values of various sandstone materials (between around 0.25 and 0.6) (Hale and Shakoor, 2003; Khanlari and Abdilor, 2015), it is perceived that the MICP specimens are more vulnerable to the FT actions i.e. the values range between 0.75 – 1.0. It should be noted, as the UCS of the 13% cemented specimens were unable to measure, the SDR_{FT} values are assumed to be 1.0. At the same time, the SDR_{FT} values decrease with the increase in cementation level (Fig. 4(b)), which is relatively express the similar tendency observed for SDR_{WD} (Fig. 4(a)). It is very clear that the calcium carbonate bonds at contact points had a vital role against FT damage. When the specimens are cemented heavily (22% of $CaCO_3$), the particle connections become stronger, which provide significant resistance to the tensile stresses developed during the freezing of porewater. However, the formation of microcracks and ruptures at sand-calcium carbonate tended to decrease the mechanical behaviors of the MICP specimens.

4. CONCLUSIONS

Experimental work presented in this paper investigated the physical and mechanical characteristics of MICP treated slope soil subjected to WD and FT cyclic actions. Based on the results, it can be concluded that the damages were higher to the specimens subjected to FT cycles compared to those subjected to WD cycles. The FT induced erosion is highly reliant on the level of

cementation. The increase in precipitated calcium carbonate content substantially increased the aggregate stability of specimens under the exposure of FT cycles. Relatively a similar tendency was observed to WD responses; however, the WD induced erosion is much less than that experienced under FT. SDR values suggest that the MICP specimens underwent severe degradation under FT cycles (over 75%). Comparatively, the reduction of UCS is low under WD cycles (between around 30 – 45%). Under both WD and FT cycles, the strength deterioration is likely to be in a relationship with the precipitated calcium carbonate content.

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